इंटरनेट मानक

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Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

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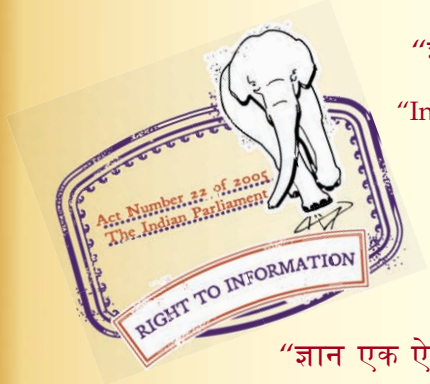
Mazdoor Kisan Shakti Sangathan
"The Right to Information, The Right to Live"

"पुराने को छोड नये के तरफ"
Jawaharlal Nehru
"Step Out From the Old to the New"

IS 5182-22 (2004): Method for measurement of air pollution, Part 22: Lead [CHD 32: Environmental Protection and Waste Management]



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"ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है"
Bhartṛhari—Nītiśatakam
"Knowledge is such a treasure which cannot be stolen"

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भारतीय मानक

वायु प्रदूषण मापन — पद्धतियां

भाग 22 सीसा

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 22 LEAD

ICS 13.040.20

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

December 2004

Price Group 5

FOREWORD

This Indian Standard (Part 22) was adopted by the Bureau of Indian Standards, after the draft finalized by the Environment Protection and Waste Management Sectional Committee had been approved by the Chemical Division Council.

Air pollution, in modern days, is one of the challenging environmental problems, faced by modern society. The damaging effects of pollutants, have caused much concern to citizens. Lead is one of such a pollutant, which is directly responsible for many present-day diseases and body-poisoning. Lead in ambient air is generated mostly by industrial, vehicular, mining and smelting activities. Therefore, developing an authentic method for its measurement, is very important.

In the preparation of this standard considerable assistance has been derived from ASTM D 3112.

The composition of the Committee responsible for formulation of this standard is given at Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS FOR MEASUREMENT OF AIR POLLUTION

PART 22 LEAD

1 SCOPE

This standard (Part 22) prescribes two methods for determination of lead in ambient air:

a) Colorimetric (Dithizone) method, and

b) Atomic absorption spectrometric (AAS) method.

2 REFERENCES

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
4167 : 1980	Glossary of terms relating to air pollution (*first revision*)

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given in IS 4167 and the following shall apply.

3.2 Particulate Lead — That collected on an efficient filter.

3.3 Vaporous Lead — That which will pass through a 0.45 µm membrane filter or its equivalent and includes various tetra alkyl lead compounds or their partially decomposed products or both.

4 COLORIMETRIC (DITHIZONE) METHOD

4.1 Principle

4.1.1 This method involves separate measurement of particulate and vaporous lead.

4.1.2 The sample of air is drawn through a sampling train consisting of a 0.45 µm membrane filter or its equivalent and then through a special sampling tube containing activated carbon. A sample shall be collected at a flow rate of 1 to 1.5 litres per minute continuously for 8 h. A plastic body or glass tube type rotameter (0.5 cpm range) may be used either on or off line for measuring flow and its variation on filter. The particulate lead sample is digested with nitric acid and perchloric acid and dissolved lead is determined by colorimetric dithizone procedure.

4.1.3 For determination of vaporous lead, the activated carbon is extracted with hydrochloric acid and nitric acid for at least 16 h at a temperature of 90° to 100°C. The carbon is removed by filtration and lead is determined in the filtrate by colorimetric dithizone method.

4.2 Range

The method is suitable for vaporous lead in ambient air at concentrations below 0.5 µg/m^3 and particulate lead in air at concentrations of 0.01 to 10.0 µg/m^3.

4.3 Interferences

Metals like bismuth, stannous tin, monovalent thallium and indium interfere.

4.4 Apparatus

4.4.1 *Absorption Cell* — 200 ml modified absorption cells as shown in Fig. 1.

4.4.2 *Cell Compartment Cover* — A special cell compartment cover is required for use with the 200 ml modified absorption cell when using a spectrophotometer.

4.4.3 *Filter and Filter Holder*

4.4.4 *Spectrophotometer*

4.4.5 *Activated Carbon Scrubber — See* Fig. 2.

4.4.6 *Gas Meter* — To measure up to about 28 litres of air per minute.

4.4.7 *Vacuum Pump* — Capable of drawing about 28 litres of air per minute.

4.4.8 *Automatic Dispensing Burette*

4.4.9 *Electrical Tapes* — Elastic type, to seal glass joints of activated carbon scrubber.

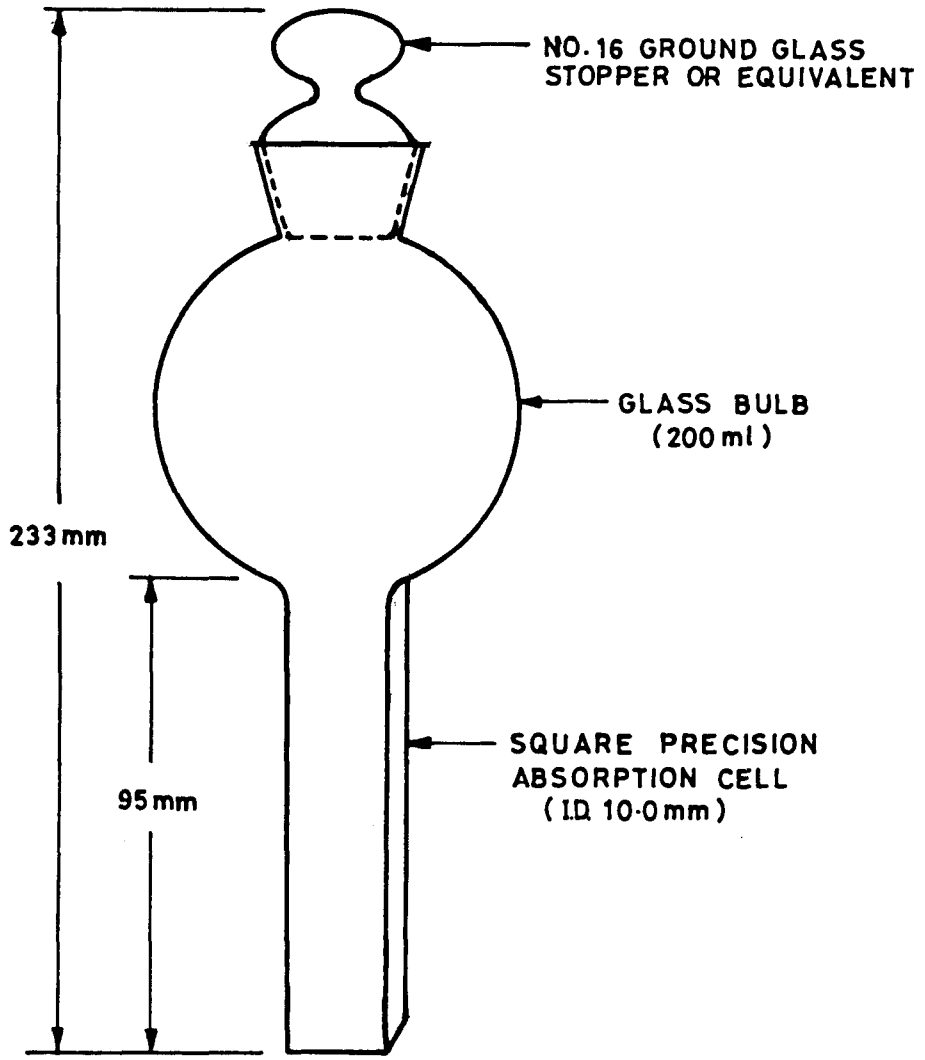

FIG. 1 MODIFIED ABSORPTION CELL

4.5 Reagents

4.5.1 *Quality of Reagents*

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4.5.2 *Activated Carbon* — 20 to 50 mesh or equivalent.

4.5.3 *Buffer Solution* — Dissolve and dilute 400 g of dibasic ammonium citrate, 10 g of hydroxylamine hydrochloride and 40 g of potassium cyanide to 1 litre with water. Mix 1 litre of citrate cyanide hydroxylamine solution with 2 litres of concentrated ammonium hydroxide.

CAUTION — KCN and its other cyanides are highly toxic to health, and they should not come in contact with acids since highly toxic and volatile HCN may be produced. Proper care should be taken in handling and disposal of cyanide containing solution.

4.5.4 *Chloroform*

4.5.5 *Ethylenediamine Tetra Acetic Acid (EDTA)* — Dissolve 5 g of EDTA in 500 ml of water.

4.5.6 *Dithizone Solution* — Dissolve 40 mg of dithizone in 1 litre of chloroform. Store at room temperature in the absence of direct light.

4.5.7 *Hydrochloric Acid*, concentrated.

4.5.8 *Standard Lead Solution I* — Dissolve 0.159 9 g of lead nitrate in about 200 ml of lead-free water. Add 10 ml of nitric acid and dilute to 1 litre with water. This solution contains 100 µg of lead/ml.

4.5.8.1 *Standard lead solution II* — Pipette out 20.0 ml

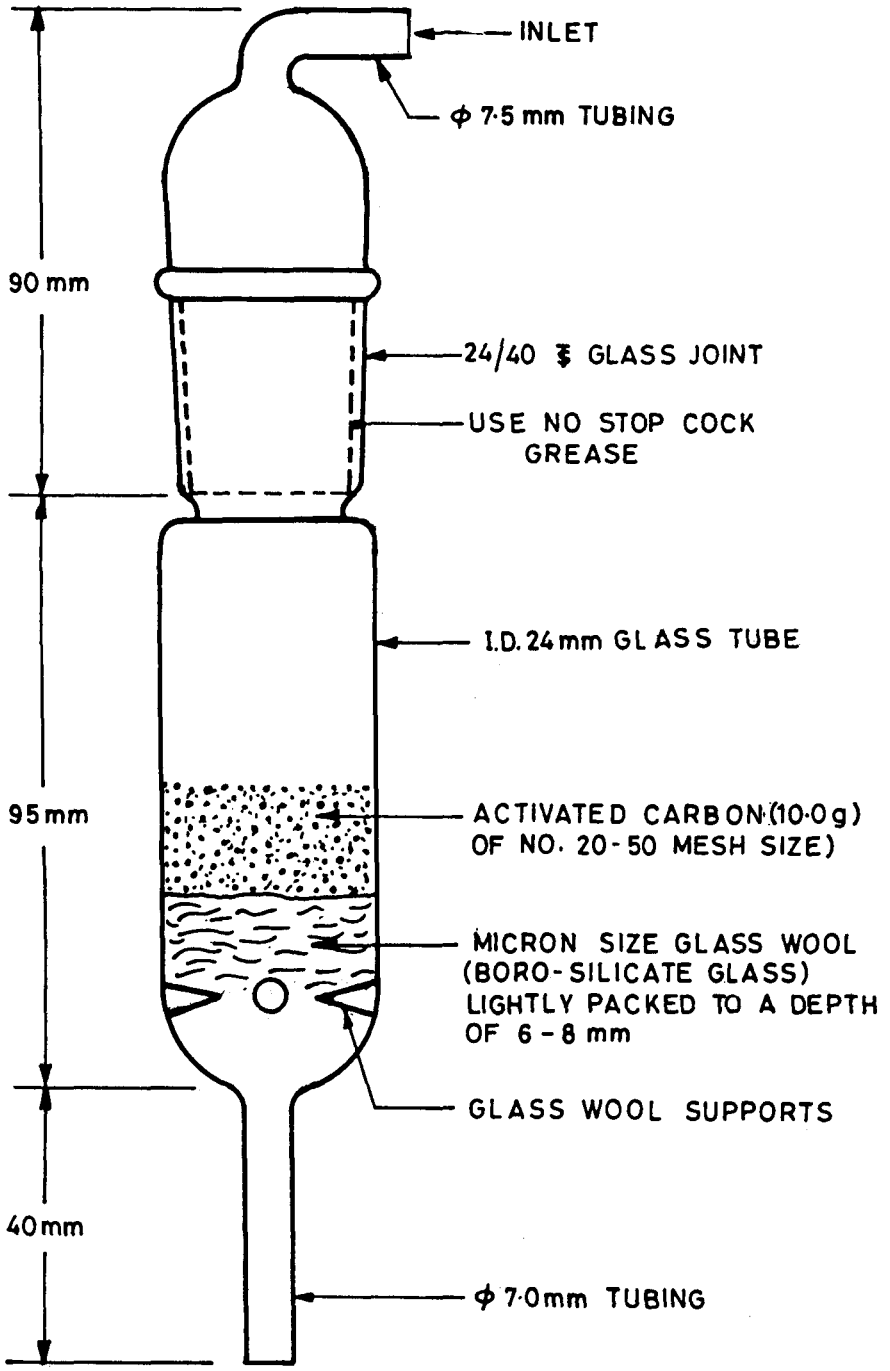

FIG. 2 ACTIVATED CARBON SCRUBBER

of Solution I (*see* **4.5.8**) and dilute to 1 litre with water. This solution contains exactly 2 µg of lead/ml.

4.5.9 *Nitric Acid*, concentrated.

4.5.10 *Dilute Nitric Acid* — Dilute 200 ml of concentrated nitric acid to 1 litre with water.

4.5.11 *Nitric Perchloric Acid Solution* — Mix 300 ml of concentrated nitric acid with 200 ml of perchloric acid (72 percent).

4.5.12 *Reducing Solution* — Weigh 20 g of potassium cyanide, 40 g of dibasic ammonium citrate, and 200 g of anhydrous sodium sulphite and dilute to 1 litre with water. Add 600 ml of concentrated ammonium hydroxide solution. Prepare this solution in a well ventilated hood.

4.6 Procedure

4.6.1 *Calibration*

Add 20 ml of nitric acid (*see* **4.5.10**), 25 ml of water and 50 ml of buffer solution to a 200 ml modified absorption cell, mix and cool to room temperature. Add 10 ml of dithizone solution and shake the mixture vigorously for 30 s. Insert the modified absorption cell into the spectrophotometer and measure the absorbance of the lower layer at 510 nm wavelength using air as reference. Add 10 ml of standard lead Solution II, shake the mixture vigorously again for 30 s and read the increase in absorbance due to the addition of known quantity 20 µg of lead. Add another 10 ml portion of standard lead Solution II, shake the mixture vigorously for 30 s and read the increase in absorbance due to the further addition of amount 20 µg of lead. Run a standard sample daily to ensure accuracy. The calibration factor, E for lead dithizonate in 10 ml of chloroform when read in spectrophotometer at 510 nm and in a 1 cm light path is approximately 35.

4.6.2 Disconnect the carbon scrubber from the sampling train and remove the tape from the 24/40 glass joint. Remove the male glass joint from the scrubber and pour the activated carbon from the scrubber into a 500 ml Erlenmeyer flask. Add 25 ml of concentrated hydrochloric acid to the carbon in the flask, swirl and then add 75 ml of concentrated nitric acid and swirl to mix, digest overnight on a hot plate or steam bath at 90° to 100°C. Add 30 ml of concentrated nitric acid to the low volume or nearly dry residue. Heat for 1 h, add approximately 100 ml of water and mix well. Allow the mixture to stand at room temperature for about 2 h. Decant the supernatant liquid through a thin rapid filter paper, hardened to great wet strength, into another 500 ml Erlenmeyer flask. Rinse the residue carbon on the filter with three portions of water. Discard the filter paper and carbon. Add 10 ml of nitric perchloric acid solution to the acid extract and heat on a hot plate to fumes of perchloric acid. If all carbon is not oxidized and solution has gone to dryness, add an additional 5 ml portion of nitric perchloric acid solution and heat to fumes of perchloric acid. To the slightly cooled perchloric acid sample, add approximately 25 ml of water from a wash bottle while washing down the sides of the Erlenmeyer flask, and add 20 ml of nitric acid. Allow approximately 30 min for complete solution of the sample and transfer the mixture to the 200 ml modified absorption cell. To the sample in the modified absorption cell, add 50 ml of buffer solution and 100 ml of reducing solution, mix and allow approximately 15 to 20 min for complete reduction of sample. Add 10 ml of dithizone solution and shake the mixture vigorously for 30 s. Insert the modified absorption cell into the spectrophotometer and measure the absorbance of the lower layer at 510 nm wavelength using air as reference. Add 5 ml of disodium EDTA solution to the modified absorption cell, shake it vigorously for approximately 90 s and measure the absorbance of the layer from 1 to 5 min after the two layers separate. The difference between the two absorbance readings represents the quantity of lead present in the sample. If a large quantity of lead is present in the sample when attempting to make the first absorbance reading as above, add an additional 10.0 ml portion of dithizone solution to sample, shake it vigorously for 30 s and measure the lead as previously described for 10 ml of dithizone solution.

4.6.2.1 Use the following procedure for particulate lead. Remove the membrane filter from the filter holder. Place it with the exposed side down in a 150 ml beaker and add 10 ml of nitric perchloric acid solution. Digest on the hot plate to fumes of perchloric acid and until all the dark carbonaceous material has oxidized. Add 20 ml of nitric acid, mix, crush filter with a glass rod and allow to cool.

4.6.2.2 Filter the sample from the 150 ml beaker through a thin, rapid filter paper, hardened to great wet strength directly into a lead-free 100 ml glass stoppered volumetric flask. Rinse the glass fibre in the filter with three 20 ml portions of water, make up to the 100 ml mark, stopper the volumetric flask and mix well.

4.6.2.3 Pipette a suitable aliquot, 25 ml of water, 50 ml of buffer solution, mix and cool to room temperature. Add 10.0 ml of dithizone solution and shake the mixture vigorously for 30 s. Insert the modified absorption cell into the spectrophotometer and measure the absorption of the lower layer at 510 nm wavelength using air as reference. Add 5 ml of disodium EDTA solution to the modified absorbance cell, shake it vigorously for about 90 s and measure the absorbance of the lower layer as in **4.6.2**. The difference between the two absorbance readings represents the quantity of lead present in the aliquot. If the initial absorbance reading is greater than 2, add additional 10 ml portions of dithizone solution to the sample before the addition of EDTA solution to dilute lead dithizonate colour and repeat the absorbance measurements after adding EDTA solution as described above.

When unexpectedly high values are obtained or when large quantities of bismuth, thallium, tin and indium are suspected, the lead dithizonate present in 10 ml of the dithizone-chloroform solution is not discharged with EDTA reagent. Instead lead dithizonate is compared with a blank made up by starting with **4.6.2.3**, but without adding the sample aliquot. The correct absorbance of the sample at 510 nm wavelength is obtained by subtracting the absorbance of the blank from the absorbance of the sample.

Interferences are detected by converting the lead dithizonate to an aqueous solution of lead nitrate and again measuring by dithizone at high *p*H. The second extraction to high *p*H will again remove approximately 90 percent of the interfering ions. Carry out determination as follows:

Transfer the entire contents of the 200 ml absorption cell to a lead free, 250 ml separatory funnel. Drain the chloroform lead dithizonate solution into a lead-free, 125 ml separatory funnel. Rinse the 200 ml absorbance cell with 5 ml of pure chloroform and transfer it to the 250 ml separatory funnel. Vigorously shake the 250 ml separatory funnel and drain the 5 ml of chloroform lead dithizonate solution into 125 ml separatory funnel. To the combined chloroform lead dithizonate solution in the 125 ml separatory funnel add 25 ml of water and 20 ml of nitric acid. Shake the stoppered 125 ml separatory funnel for 15 s and discard the chloroform solution. Transfer the 45 ml of nitric acid containing all the lead to the original 200 ml absorption cell, add 50 ml of buffer solution and 10.0 ml of dithizone, shake as before, and again measure the absorbance of the lead dithizonate solution. Correct for a blank carried through all the steps of the double extraction procedure. If the corrected absorbance is near that found in measurement of the original lead dithizonate solution, no interference was present in the original sample. If the absorbance of the second extract is 10 percent or more, below that of the original extract, interferences in higher quantities may be present.

4.7 Calculation

4.7.1 Calculate the calibration factor, F as follows:

$$F = \frac{X}{(Y-Z)}$$

where

X = quantity of lead in calibration sample,

Y = absorbance after adding lead, and

Z = absorbance before adding lead.

4.7.2 Calculate the vaporous lead (C_V) in µg/m³ of air as follows:

$$C_V = [(A-B)-(C-D)] \times F \times \frac{(1\,246.6)}{H} \times \frac{G}{10}$$

4.7.3 Calculate particulate lead (C_O) in µg /m³ of air as follows:

$$C_O = [(A-B)-(C-D)] \times F \times \frac{(1\,246.6)}{H} \times \frac{G}{10} \times \frac{(100)}{F}$$

where

A = sample absorbance before EDTA treatment;

B = sample absorbance after EDTA treatment;

C = blank absorbance before EDTA treatment;

D = blank absorbance after EDTA treatment;

F = volume of aliquot removed from the 100 ml glass stoppered volumetric flask, in ml;

G = volume of dithizone solution, in ml; and

H = volume of air, in m³.

5 ATOMIC ABSORPTION SPECTROMETRIC (AAS) METHOD

5.1 Principle

This method is based on acid digestion and atomic absorption spectrometry for the chemical analysis of lead samples collected on filters from ambient air. The method is applicable to ambient air samples with particulate lead contents, such that the amount of deposited particulate lead collected on the filter is greater than 1 µg if the final determination is made by flame atomic absorption spectrometry. Final determination by graphite furnace atomic absorption spectrometry allows measurement of quantities of less than 1 µg, but is only applicable after experimental validation of detection limits.

5.2 Detection Limit

The minimum detection limit of the method with flame atomic absorption spectrometry is 0.1 µg/ml. This corresponds to a concentration of 5 µg/m³ when sampling is done for 8 h at an average flow rate of 1.1 m³/min and a final sample solution of 25 ml.

5.3 Interferences

The method may not be suitable for samples with high ratios between an interfering element and lead. The nature and extent of interference depends on whether flame or graphite furnace atomic absorption spectrometry is used. The only major spectral interference, which is likely to occur, is due to antimony when a wavelength of 217 nm is used. Where high concentrations of sodium are present in sample solutions, efficient correction of non-specific background absorbance is essential.

5.4 Apparatus

5.4.1 *High Volume Sampler or Respirable Dust Sampler*

The high volume sampler or Respirable Dust Sampler (HVS attached with cyclone separator) shall be capable of drawing air through a portion of a clean glass fibre of 20 cm × 25 cm size with an effective area of not

less than 400 cm^2 at a flow rate of 1 m^3/min with a permissible variation of 0.3 m^3/min over 24 h.

5.4.2 *Hot Plate (Thermostatically Controlled)*

5.4.3 *Sampling Medium*

Filters for collection of particulate matter shall be of glass fibre type of 0.45 micron pore size. Unexposed filters shall have lead content considerably lower than minimum quantity measurable by the AAS procedure used.

5.4.4 *Atomic Absorption Spectrometer*

Set up and operated according to the manufacturers instruction and equipped with a burner for use with an air/acetylene flame and/or a graphite furnace with auto injection, a hollow cathode lamp or an electrodeless discharge lamp, and a capability for correction of non-specific attenuation by using a deuterium lamp or Zeeman background correction system.

5.5 Reagents

5.5.1 *Distilled or Deionized Water* — With a lead content less than 0.01 µg/ml and electrical conductivity less than 0.2 ms/m (2 µs/cm).

5.5.2 *Nitric Acid (HNO_3), Concentrate* — Nitric acid having a specific gravity of 1.42 g/ml redistilled with lead content less than 0.01 µg/ml.

5.5.3 *Nitric Acid, Dilute* — Approximately 0.1 mole/ litre. Add 10 ml of concentrated nitric acid to 500 ml of water and dilute to 1 litre with water.

5.5.4 *Lead Standard Solution (1 000 µg Pb/ml)* — Dissolve 1.598 g of lead nitrate $Pb(NO_3)_2$, previously dried to constant mass at 110°C and cooled in a desiccator, in dilute nitric acid. Quantitatively transfer the solution to 1 000 ml volumetric flask and make up to the mark with nitric acid.

5.5.5 *Hydrogen Peroxide (30 %)* — Approximately 300 g/litre with a lead content less than 0.01 µg/ml.

5.6 Procedure

5.6.1 *Sampling Method*

Samples of airborne particulate matter are collected on glass fibre filters of 20 mm × 25 cm size by means of high volume sampler at an average flow rate of 1.1 m^3/min. Sampling time may range between 8 to 24 h.

5.6.2 *Digestion Procedure for Filters*

The exposed glass filters are cut into pieces by means of clean stainless steel scissors and transferred into a 250 ml beaker. To the beaker is added 6 ml of concentrated nitric acid, 4 ml of hydrogen peroxide (30 %) and 50 ml of distilled water. Cover with a watch glass and heat on a hot plate until most of the acid has evaporated. Repeat this addition of nitric acid, hydrogen peroxide and distilled water followed by evaporation, at least twice. Then continue to heat until the residue is barely dry and a white ash appears. Do not bake the residue. If the residue ignites, discard the sample, as lead would have been lost. Dissolve the residue in 5 ml of concentrated nitric acid. Filter the digest, with repeated small washings of nitric acid into a 25 ml volumetric flask and make up to mark with dilute nitric acid.

5.6.3 *Determination of Lead by AAS*

5.6.3.1 *Preparation of calibration solutions*

Prepare a calibration blank solution and atleast five calibration solutions to cover the range of expected concentrations of the test solutions, within the linear operating range of the atomic absorption spectrometer by dilution of the lead standard solution.

5.6.3.2 *Setting up of the instruments*

Set up the atomic absorption spectrometer to the manufacturer's instructions, and optimize the setting of parameters including lamp current and mono-chromatic slit width. For flame atomic absorption spectrometry, optimize burner height, fuel and oxidant flow rates and nebuliser flow rate. For graphite furnace atomic absorption spectrometry, establish the optimum temperature programme to avoid losses of lead, especially during the ashing phase of the temperature programme. Do not use graphite furnace atomic absorption spectrometry without auto-injection. In all cases, correction for non-specific attenuation shall be used.

5.6.3.3 *Plotting of calibration curve*

Prepare a calibration graph by plotting the absorbance of each calibration with respect to the absorbance of the calibration blank solution, *versus* the concentration of lead in the calibration solutions, in micrograms per millilitre (or if graphite furnace atomic absorption spectrometry is used, in microgram per litre).

5.6.3.4 *Blank factor*

Analyze at least one unexposed filter with each batch of exposed test filters.

5.6.3.5 *Spectrometric measurement*

Determine the concentration of lead in the digested sample solutions using either flame or graphite atomic absorption spectrophotometer, by measuring the absorbance at a wavelength of 217 nm or 283.3 nm, with correction for non-specific attenuation. The sample concentration is related to the absorbance, and

can be determined from the appropriate calibration graph. Use only linear part of the calibration curve and dilute the test solutions whose response falls outside this region with an appropriate volume of dilute nitric acid. Record the dilution factor used. For the graphite furnace procedure, use the same final sampling volume for both analysis and calibration.

5.6.3.6 *Blank solutions*

Analyze all of the blank solutions and subtract the mean lead concentration of the blank solutions from the lead concentration of the sample solutions. Where sample solutions are diluted into the linear operating range of the atomic absorption spectrometer, an equivalent dilution shall be made of the blank solutions, and the mean lead concentration of this diluted solution subtracted from the lead content of the diluted sample solutions. Use standardized statistical methods to determine the detection limit based on the standard deviation of the lead concentration in a minimum of six solutions obtained by dissolution blank filters.

5.7 Calculation

Express the mass concentration of lead in micrograms per cubic meter in air sample to the nearest 0.1 μg /m^3 using equation:

$$Pb\,(Con) = \frac{[Pb\,(Sm) - Pb\,(Bl)] \times V_t}{V}$$

where

$Pb\,(Con)$ = mass concentration of particulate lead, in μg/m^3;

$Pb\,(Sm)$ = concentration of lead, in μg/ml, in sample solution;

$Pb\,(Bl)$ = concentration of lead, in μg/ml, in blank solution;

V_t = total volume of digested sample in ml; and

V = volume of air sampled, in m^3.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

Environment Protection and Waste Management Sectional Committee, CHD 32

Organization	*Representative(s)*
In personal capacity (*40/14, C.R. Park, New Delhi-110019*)	PROF DILIP BISWAS (***Chairman***)
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BIS Directorate General	DR U. C. SRIVASTAVA, Director & Head (CHD) [Representing Director General, BIS (*Ex-officio*)]

Member-Secretary
SHRI N. K. PAL
Director (Chemical), BIS

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc: No. CHD 32 (9610).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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